

Removal of Octylphenolethoxylate from Waste Water using Carbonized and Non Carbonized Agro Waste of *Typhalatifolia*

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Abstract

Removal of higher concentration of nonionic surfactant Octylphenolethoxylate with 10 moles of ethylene oxide (OPE-10) trade name, Octylphenolethoxylate from industrial effluents, using the carbonized stem of aquatic weed *Typhalatifolia*, has been discussed in the present investigation. Carbonization of *Typhalatifolia* stem was done in the laboratory by using Muffle furnace instrument at temperature 500°C. The removal of surfactant is calculated by U-V spectrophotometer using cobalt thiocyanate active substances (CTAS) method. Various concentrations of surfactant solutions in the range from 200ppm to 1000ppm have been prepared for extraction work.

Optimization of the experiments is done by calculating variables – shaking time, doses of absorbent bed and concentration of reagents. Degree of adsorption is validated and compared with isotherm models of Langmuir adsorption isotherms, Freundlich adsorption isotherms, Temkin adsorption isotherms, Dubinin-Radushkevich adsorption isotherms and Harkin Jura isotherms. R^2 value among all isotherms best suited is Freundlich adsorption isotherm. SEM images of non-carbonized and carbonized agro-waste are also analyzed and compared. The variance of ANOVA was considered significant at $p = 0.25$.

Keywords: Adsorption Isotherms, Removal of surfactant, Industrial effluents., Dubinin-Radushkevich, Freundlich, Harkin-Jura, Temkin.

Introduction

Surfactants are a group of chemicals which are widely used for detergent and cleaning purpose. Surfactant reduces interfacial tension with respect to an adjacent phase, therefore lies at the heart of interfacial chemistry. Surfactants are widely used in pharmaceutical industries, agriculture, cosmetics etc⁴. After use, the role of surfactants in the environment is ambiguous. It creates problems in the wastewater as the presence of surfactants in effluents hinders the transfer of oxygen from the atmosphere to the water in the process of aeration. They reduce the oxygen transfer efficiency to 15%¹⁰.

They trap the colloidal particles and keep them on the surface, thus reducing the efficiency of coagulation. Once

they find a way into surface of sub-surface waters, they join to food chain¹³. They accumulate in body and exhibit long term disorders like carcinogenicity, mutagen city, fertility loss, loss of potency, allergy etc. in long term. They may bio-accumulate and their manufacture processes and by-products can be environmental harmful.

Yuan et al¹⁸ from China has discussed that surfactants have broad applications in manufactured goods and industry. A bulky amount of surfactant containing wastewater is discharged into the environment resulting in harming aquatic life, Therefore, it is important to observe and control emissions of surfactants in environmental water.

The methods available in the literature for removal of surfactants are mostly chemical methods in which $\text{TiO}_2/\text{Al}_2\text{O}_3$ is the photo catalyst for removal of surfactant concentration from water sample^{1,6}. Removal of surfactant from industrial wastewaters is done by the coagulation, flocculation process using FeCl_3 and alumina^{2,7}. TiO_2 is used as photo catalyst for removal of surfactants¹⁷. Surfactant can be removed by membrane technique in which affecting factors are trans-membrane pressure and pore size⁵. Partially aerobically biological degradation method is an option for degradation of surfactants from sewage sludge¹⁵. Activated charcoal and agricultural waste can be used for removal of cationic surfactants⁸.

Generally, the removal of detergents from wastewater is favored by chemical methods. But these methods simply transfer the problem from one phase to another. In present study, removal of nonionic surfactant Octylphenolethoxylate from wastewater is used. The absorption of surfactant is calculated by UV-Spectrophotometer and validated by adsorption isotherm. The efficiency of particle size of absorbent is discussed by SEM technique and statically proved by Anova. So, removal of surfactant from wastewater is to find a method which may be eco-friendly and biodegradable in nature.

Material and Methods

Preparation of stock solutions: 1N stock solution of ammonium thiocyanate (NH_4SCN) was prepared in double distilled water and stored in an amber airtight bottle. Reagents cobalt nitrate $\text{Co}(\text{NO}_3)_2$ and benzene used are Qualigens LR grade. Working solutions from 200 ppm to 1000 ppm concentrations of Triton -X 100 were prepared by the dilution method from stock solution.

Preparation of carbonized Typhalatifolia stem [TS]:

Stem of Typhalatifolia was collected from the local wetland area. Stem was washed with water and de-ionized water to remove dust, non-metallic and metallic impurities. Then it was dried in sunlight for 4 to 5 days. The dried stem of Typhalatifolia was sieved in the net in the range between 250 μ m and 150 μ m in order to raise its surface area.

Carbonization of dried Typhalatifolia stem was done in a Muffle furnace at 500°C for 4 minutes as shown in figure 1b and figure 1c. After that red-hot carbonized char was poured into ice cold water and washed twice with distilled water. Dried in the oven at 100–110°C and stored in airtight container. Figure 1a shows steps of carbonization TS.

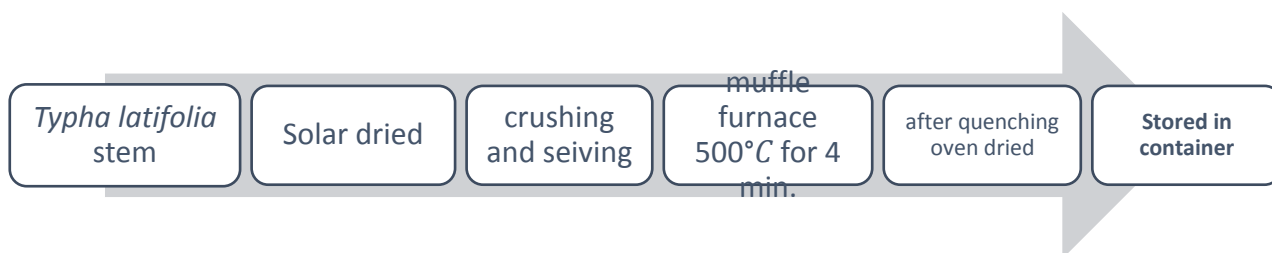


Figure 1a: Flow diagram for carbonization of Typhalatifolia stem



Figure 1b: Non Carbonized Typhalatifolia stem



Figure 1c: Carbonized Typhalatifolia stem

All experiments are measured by using PC based double beam SYSTRONICS UV-spectrophotometer model no. 2202 by formation of a coloured complex between ammonium cobalt thiocyanate reagent with surfactant compound method as reported earlier⁹.

SEM Technique: SEM analysis was analyzed by JEOL MAKE (UK) Model no.- SM6360.

Equilibrium studies: Equilibrium studies using UV spectrophotometer 1 gram carbonized and non-carbonized agro char were made for adsorption process with different concentrations of 50 ml surfactants solution up to 35 min. After filtration, treat with 20 ml of 1 N ammonium thiocyanate solution along with 0.5 ml of cobalt nitrate in an airtight beaker at 30°C. Solution was transferred in to separating funnel and allowed to stand for 15 min to settle down. After formation of two separate layers, analysis is processed for adsorption in UV spectrophotometer.

Statistical Analysis: Statistical analysis was performed by computer software SPSS. All data were subjected to one-way analysis of variance (ANOVA). Statistical Analysis ANOVA was performed on absorbance of surfactant and non-carbonized Typhalatifolia stem and surfactant and carbonized Typhalatifolia stem.

Adsorption of Octylphenoethoxylate: Adsorption of no. of moles of surfactant (Octylphenoethoxylate) by per one gram carbonized and non-carbonized agro is calculated by the formula shown in eq. (1):

$$x = Ce \cdot V \cdot Eq / 1000 \quad (1)$$

where x = No. of moles of Octylphenoethoxylate NB, V = Volume of test solution in ml and Eq= Equivalent weight of Octylphenoethoxylate in the gm.

Adsorption Isotherm

1. Langmuir Adsorption Isotherm: The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identified sites. The model assumes uniform energies of adsorption onto the surface and no transmigration of the adsorbate in the plane of the surface.^{9,14} Langmuir represented the following equation.

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 K_L C_e} \quad (2)$$

where C_e = concentration of adsorbate (mg/L-1), q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g), Q_0 = maximum monolayer coverage capacity (mg/g) and K_L = Langmuir isotherm constant (L/mg).

2. Freundlich Adsorption Isotherm: This is commonly used to describe the adsorption characteristics for the heterogeneous surface³. These data often fit the empirical equation proposed by Freundlich:

$$Q_e = k_f C_e^{1/n} \quad (3)$$

The constant k_f is an approximate indicator of adsorption capacity.

3. Temkin Adsorption Isotherm: This isotherm contains a factor explicitly taking into the account of adsorbent–adsorbate interactions. As implied in the equation, its derivation is characterized by a uniform distribution of binding energies³. The model is given by the following equation:

$$q_e = \frac{RT}{b} \ln(A_T C_e) \quad (4)$$

where A_T = Temkin isotherm equilibrium binding constant (L/g), bT = Temkin isotherm constant R = universal gas constant (8.314J/mol/K) and T = Temperature at 298K.

4. Harkin-Jura Adsorption Isotherm: This model assumes the possibility of multilayer adsorption on the surface of adsorbent having a heterogeneous pore distribution¹⁶. Mathematically, this model can be expressed as in equation (5):

$$\frac{1}{q_2} = \frac{B}{A} - \left(\frac{1}{A}\right) \log C_e \quad (5)$$

where B and A are constants

5. Dubinin–Radushkevich Adsorption Isotherm: The Dubinin-Radushkevich isotherm model is another empirical model which is initially formulated for the adsorption process following a pore filling mechanism¹². It is generally applied to express the adsorption process occurred onto both homogeneous and heterogeneous surfaces. The non-linear expression of Dubinin-Radushkevich isotherm model can be illustrated as equations (6) and (7):

$$q = q_s \exp(-K_{DR} \varepsilon^2) \quad (6)$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e}\right) \quad (7)$$

where R = universal gas constant (8.314J/mol/K), T = Temperature at 298K and C_e is the equilibrium constant.

Thermodynamic Parameters: In environmental engineering practice, both energy and entropy factors must be considered in order to determine which process will occur spontaneously. In Gibbs free energy change, ΔG is the fundamental criterion of spontaneity. Reactions occur spontaneously at a given temperature if ΔG is a negative quantity¹¹. The free energy of the sorption reaction,

considering the sorption equilibrium constant, K_L is given by the equation presented below:

$$\Delta G = -RT \ln K_L \quad (8)$$

where ΔG is the standard free energy change, R is the Universal gas constant 8.314 J mol⁻¹ and T is the absolute temperature.

Results and Discussion

Octylphenoethoxylate is a nonionic surfactant; it has a polyethylene oxide with hydrophilic nature, chain and an aromatic hydrocarbon with hydrophobic nature. The structural formula of octylphenoethoxylate is 4-(1,1,3,3-tetramethylbutyl)-phenyl group in figure 2.

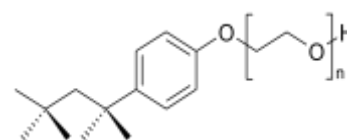
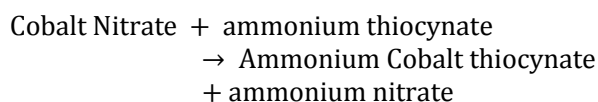


Figure 2: Structural formula of Octylphenoethoxylate

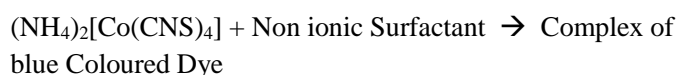
Mechanism for Spectroscopic analysis: On addition of cobalt Nitrate and a crystal of ammonium thiocyanate in extracted working solution, a pink colour is obtained in the organic layer due to the formation of ammonium cobalt thiocyanate coloured complex.

Step I



Step II

Solution of this pink coloured ammonium cobalt thiocyanate is added to a surfactant having polyethylene oxide chain with more than 6 carbon. A blue color dye is formed.



Coloured complex is measured by UV spectrophotometer at λ_{max} of 436 nm.

Statistical Analysis by Anova: The data of absorbance was subjected to one-way analysis of variance (ANOVA). The Statistical Analysis ANOVA is performed on absorbance of (a) surfactant and noncarbonized Typhalatifolia stem (b) surfactant and carbonized Typhalatifolia stem. The variance of Anova is considered significant at $p = 0.25$.

Adsorption Isotherms: Adsorption process can be represented by adsorption isotherms. It is the graph between the amounts of adsorbate adsorbed on the surface of adsorbent at constant temperature.

1. Langmuir Adsorption Isotherm: The values q and K_L were calculated from the slope and intercept by Langmuir graph of $1/q$ versus $1/C_e$. The fundamental features of the Langmuir isotherm may be uttered in terms of equilibrium parameter R_L which is a dimensionless constant or equilibrium parameter¹⁶.

$$R_L = \frac{1}{1+(K_L C_i)} \quad (9)$$

where C_i = initial concentration, K_L = Langmuir Constant. R_L value indicates the adsorption nature. If $R_L > 1$, unfavorable, if $R_L = 1$, it is linear and if $0 < R_L < 1$, it is favorable. From the data interpreted in table 1, value of R_L is greater than 0 but lesser than 1, so it favors Langmuir isotherm.

For surfactant verse non carbonized values from table 1 are $Q_{Max} = 0.56 \text{ mg/g}$, $K_L = 1984 \text{ L/mg}$, $R_L = 2.5 \times 10^6$ indicates that the equilibrium sorption is favorable and R^2 -value 0.903 indicates the sorption data good fitted into Langmuir Isotherm model.

For surfactant verse carbonized values from table 1 are $Q_{Max} = 0.18 \text{ mg/g}$, $K_L = 6250 \text{ L/mg}$, $R_L = 0.79 \times 10^6$ indicates that the equilibrium sorption is favorable and R^2 -value 0.913 indicates the sorption data good fitted into Langmuir Isotherm model.

2. Freundlich Adsorption Isotherm: Langmuir's model was a theoretical construct while the Freundlich isotherm is empirical. If the value of n is in the range of 2–10, it indicates good;¹⁶ if n value is 1–2, it is moderate, if below than 1, then it is poor adsorption. In our study n value is moderate.

For surfactant verse non carbonized values of adsorption intensity are $1/n = 0.74$, while $n=1.35$ it is moderate indication for the sorption of OPE-10 onto agro-char. R^2 value found 0.951 is supportive for the statement.

For surfactant verse carbonized values of adsorption intensity are $1/n = 0.42$, while $n=2.33$ it is good indication for the sorption of OPE-10 onto agro-char. R^2 value found 0.951 is supportive for the statement.

3. Temkin Isotherm: This equation can be linear as:

$$q_e = B \ln A + B \ln C_e \quad (10)$$

where $B = \frac{RT}{bT}$, A = binding constant (L/g), b_T = Temkin constant, R = universal gas constant (8.314 J/mol/K), T = Temperature at 303⁰K and B = Constant related to heat of sorption (KJ/mol).

For surfactant verse non carbonized values estimated are, $A = 0.116 \text{ L/g}$, $B = 3222 \text{ J/mol}$; this is an indication of the heat of sorption and also indicating a physical adsorption process. We obtain the value of bT as 0.781 KJ/mol, this constant is

related to the heat of adsorption, these constants are calculated from the plot q verse $\ln C_e$ and tabulated in table 1. Coefficient R^2 values gives good co-relation 0.988.

For surfactant verse carbonized values estimated are, $A = 0.115 \text{ L/g}$, $B = 2666 \text{ J/mol}$; this is an indication of the heat of sorption and also indicating a physical adsorption process. the value of bT is 0.944 KJ/mol, this constant is related to the heat of adsorption and the coefficient R^2 value 0.992 gives good co-relation. So, it may be concluded that this model gives better agreement for the equilibrium data compared to the Langmuir, Freundlich and Harkin-Jura isotherm models.

4. Dubinin–Radushkevich Isotherm: According to this isotherm, linear graph plot is obtained by equation 7. Dubinin-Radushkevich adsorption isotherms was obtained¹². The values of regression coefficient can be calculated by using formula:

$$\ln q = \ln q_{DR} - \beta \varepsilon^2 \quad (11)$$

For surfactant verse non carbonized agro waste, values of q_{DR} and β are obtained after calculation as $252 \text{ mg} \cdot \text{g}^{-1}$ and $0.00057 \text{ mol}^2 \cdot \text{kJ}^{-2}$ respectively. ε is the Polanyi potential. This model is generally useful to determine the mean free energy of adsorption (E). $E = 29.67$. The value of linear regression coefficient R^2 is in the range of 0.936.

The values of surfactant verse carbonized values of q_{DR} and β are obtained after calculation as $797 \text{ mg} \cdot \text{g}^{-1}$ and $0.00062 \text{ mol}^2 \cdot \text{kJ}^{-2}$ value of $R^2 = 0.945$. E value 29.41 is revealing that the experimental data fitted well with the Dubinin-Radushkevich isotherm model. If the value of E is less than 8 KJ mol^{-1} , it is called physical adsorption and if the E values are between 8 and 16 KJ mol^{-1} , it is called chemical. In the present study, the E value is $29.41 \text{ KJ mol}^{-1}$ which reflects the physical sorption due to formation of chemical bonds between surfactant and the carbonized stem of *Typhalatifolia*.

5. Harkin-Jura Isotherm: This isotherm describes the multilayer adsorption on the surface of adsorbent having a heterogeneous pore substance¹². This isotherm can be expressed by this equation:

$$\frac{1}{q_2} = \frac{B}{A} - \left(\frac{1}{A}\right) \log C_e \quad (12)$$

where A and B are the Harkin-Jura constants obtained from plot $1/q_e$ vs. $\log C_e$.

The heat of adsorption decreases in magnitude with increasing adsorption for most of the adsorption systems. The value of surfactant verse non carbonize is $B = 7.63 \text{ mol g}^{-1}$, $A = 9.3 \text{ mol g}^{-1}$ and for surfactant verse carbonized $B = 6.70$, $A = 1.11$ values indicated in table 1. R^2 value of 0.823 and 0.923 is respectively for non-carbonized and carbonized agro waste with surfactant.

Results by Graphs

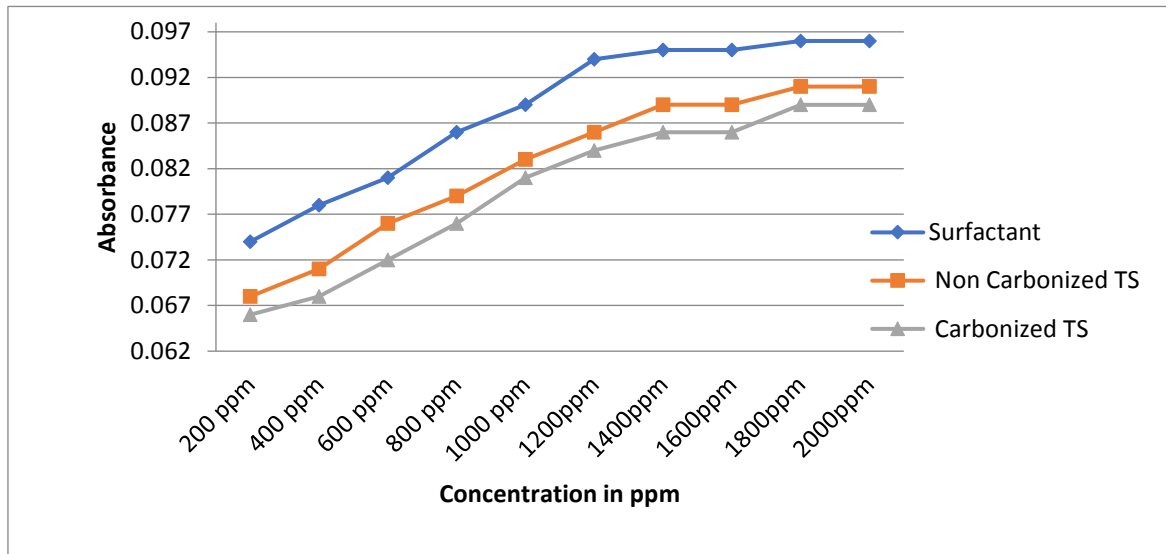


Figure 3: Graph plot among Absorbance of Surfactant and after passing by Non-carbonized TS and carbonized TS.

Surfactant verse non carbonized Typhalatifolia graphs

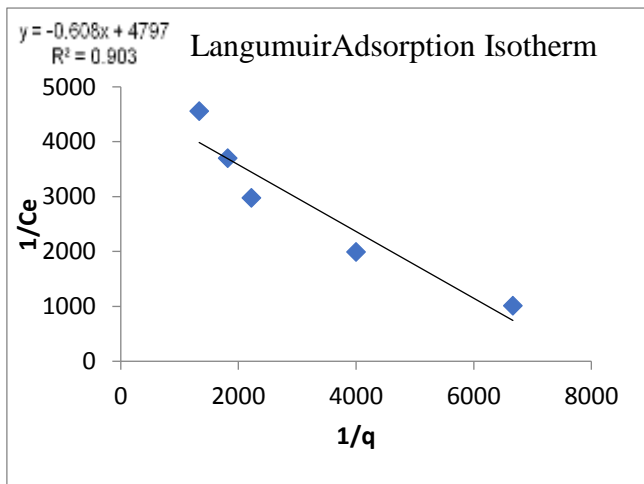


Figure 3a: The graph plot between 1/q Vs 1/Ce

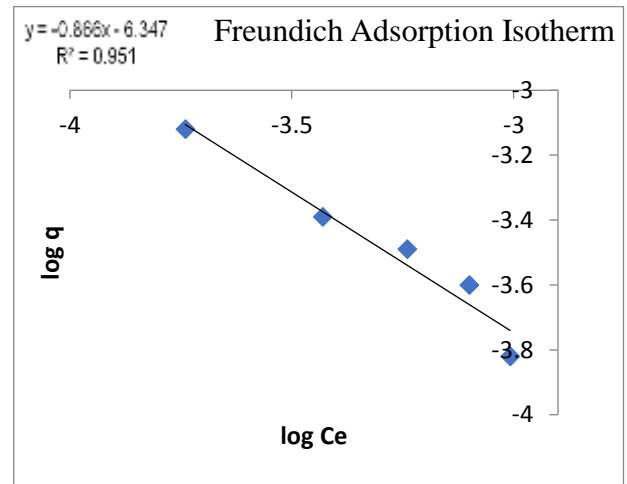


Figure 3b: The graph plot between Log Ce Vs Log q

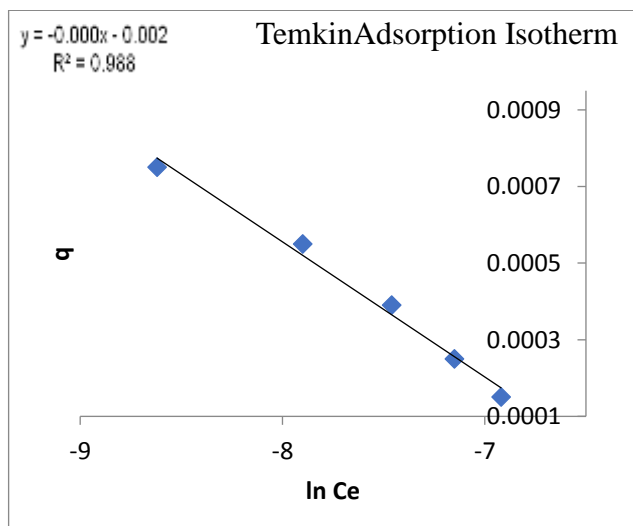


Figure 3c: The graph plot between q Vs ln Ce

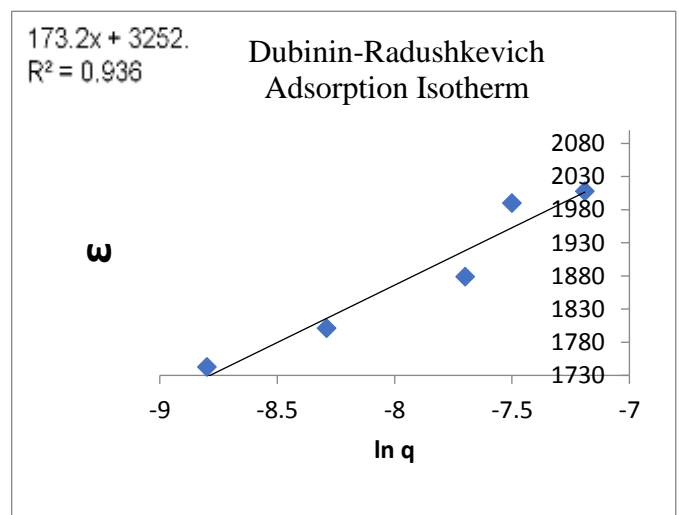


Figure 3d: The graph plot between ln q Vs ε

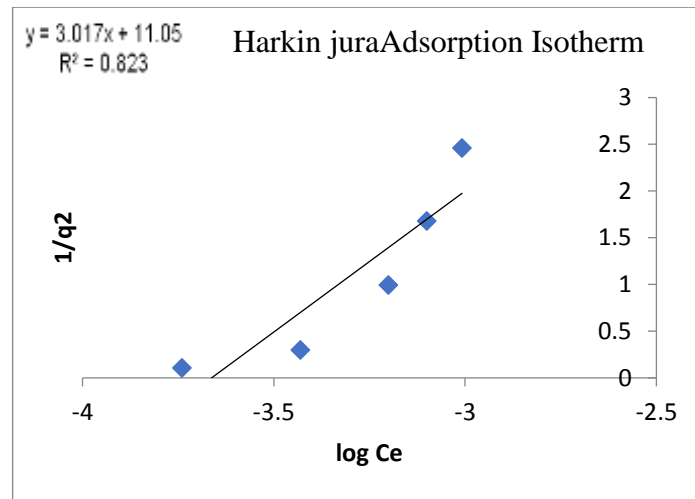


Figure 3e: The graph plot between 1/q2 Vs log Ce

Surfactant and carbonized Typhalatifolia graphs

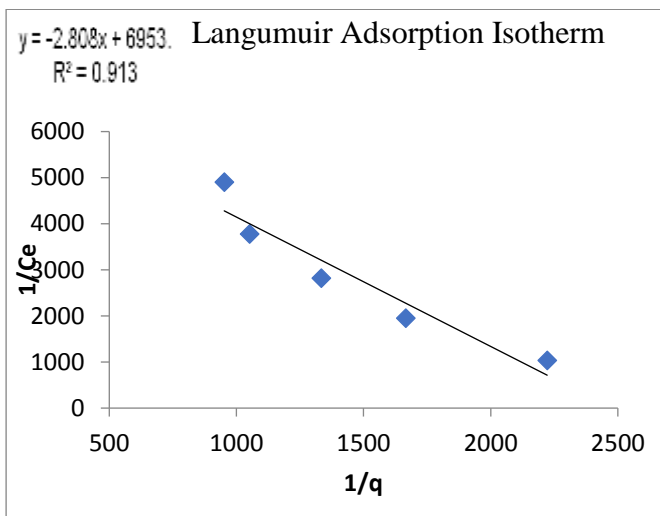


Figure 4a: The graph plot between 1/q Vs 1/Ce

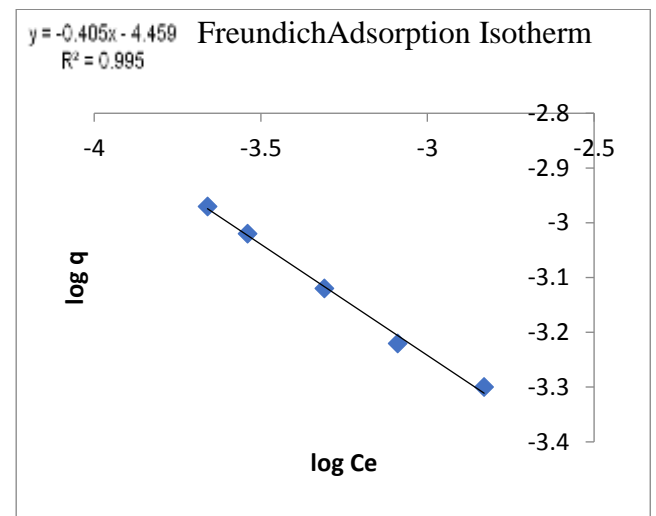


Figure 4b: The graph plot between Log Ce Vs Log q

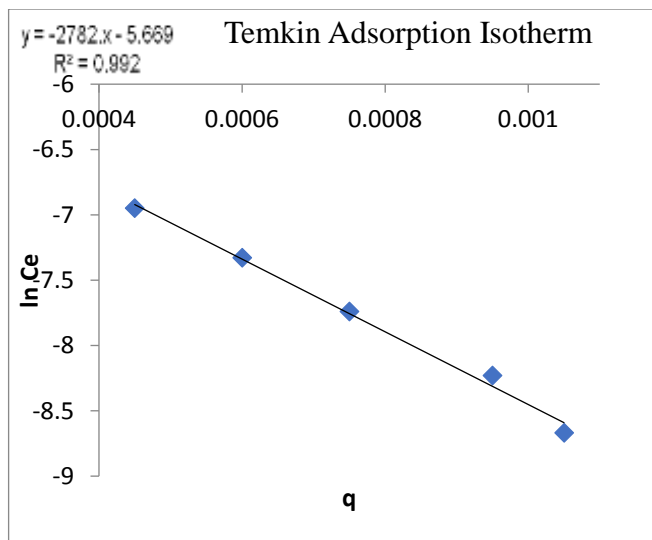


Figure 4c: The graph plot between q Vs ln Ce

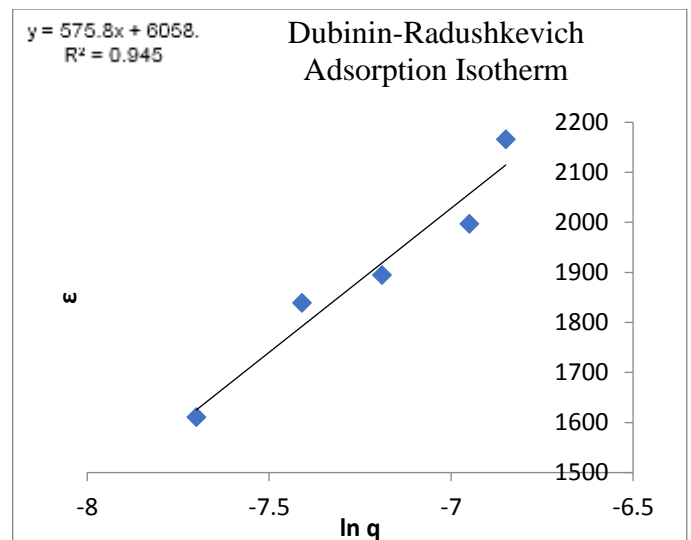


Figure 4d: The graph plot between ln q Vs ε

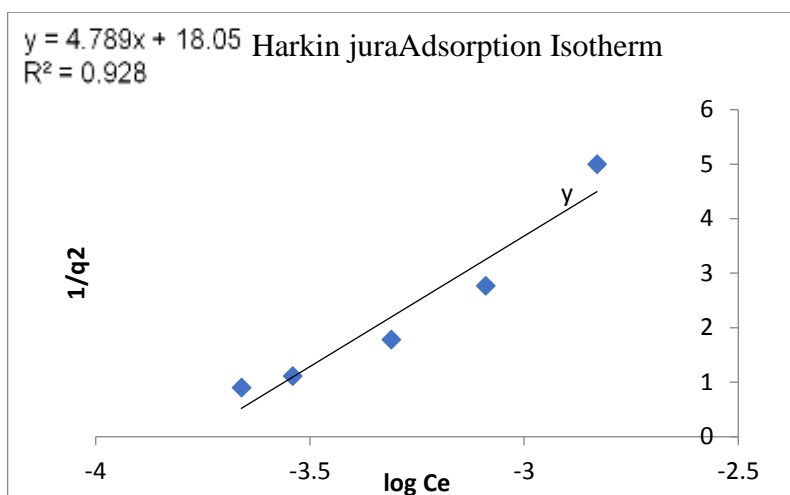


Figure 4e: The graph plot between 1/q² Vs log C_e

Table 1

Describe Adsorption Isotherm Parameter of Langmuir adsorption isotherm, Freundlich adsorption isotherm, Temkin adsorption isotherm, Harkin Jura adsorption isotherms and Dubinin-Radushkevich adsorption isotherm of Surfactant verse Non-Carbonized and carbonized agro waste

Isotherm	Parameter	Unit	Non-Carbonized Value	Carbonized Value
Langmuir	Q _{max}	Mg, g ⁻¹	0.56	0.18
	K _L	L mg ⁻¹	1984	6250
	R _L		2.5x10 ⁻⁶	0.79x10 ⁻⁶
	R ²		0.903	0.913
Freundlich	1/n		0.740	0.429
	n		1.351	2.33
	K _H	L mg ⁻¹	0.0219	0.0368
	R ²		0.951	0.995
Temkin	A	L mg ⁻¹	0.116	0.115
	B		3222	2666
	b _T	KJ Mol ⁻¹	0.781	0.944
	R ²		0.988	0.992
Harkin jura	A	mol. g ⁻¹	9.3	1.11
	B	mol. g ⁻¹	7.63	6.70
	R ²		0.823	0.923
Dubinin-Radushkevich	q _{DR}	mol. g ⁻¹	252	797
	β	(mol ² . kJ ⁻²)	0.00057	0.00062
	E	KJ Mol ⁻¹	29.67	29.41
	R ²		0.936	0.945

Thermodynamic Parameters

Gibb Free Energy: Maximum negative value of ΔG reaction has shown the feasibility of the creation.

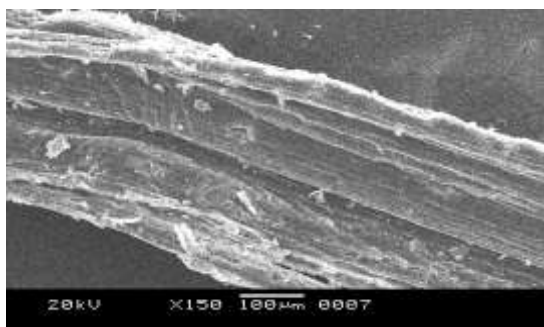
$$\Delta G = - 2.303 RT \log K_L \tag{13}$$

where T= absolute Temperature in Kelvin= 300, The value of K_L is obtained by table 2, So log K_L = 3.29.

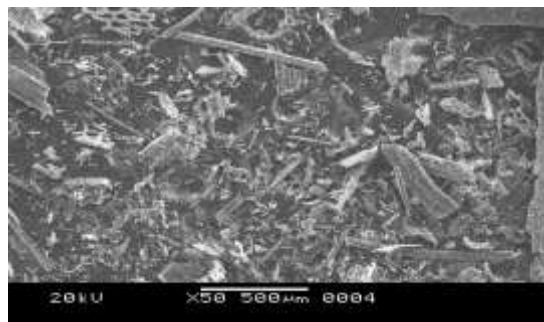
Gibb Free Energy ΔG = -19087J / Mole or -19.087KJ / Mole for non-carbonized TS.

For carbonized log KL = 3.79

Gibb Free Energy ΔG = -21988 J / Mole or -21.988 KJ / Mole for carbonized TS.



**Figure 5a: SEM image of Non-Carbonized TS
Magnification 150000**



**Figure 5b: SEM image of Carbonized TS
Magnification 50500**

SEM analysis analyzed by JEOL MAKE (UK) Model no.- SM6360: Carbonized agro-char is extremely porous carbon particle whose inner surface area is very large. This is proven by SEM analysis. This porous structure attracts and holds organic molecules as well as certain metal or inorganic molecules. Adsorption occurs due to:

- i) The pollutant has low solubility in the waste
- ii) The pollutant has bigger affinity for the carbon than for the waste and
- iii) A combination of the i and ii option.

Conclusion

In the present investigation, equilibrium sorption is carried out at an absorbance between 0.069 to 0.18. Five adsorption isotherm models were studied. The sorption data fitted into Dubinin–Radushkevich, Langmuir, Freundlich, Temkin and Harkin jura isotherms, out of which Freundlich Adsorption model was found to have the highest regression value and hence the best fit.

By the SEM technique, variance of Anova was considered significant at $p = 0.25$ and Gibbs free energy value concluded that carbonized Typhalotifora Stem is potential to be an active bio sorbent for removal of octylphenolethoxylate from its aqueous solution and industrial wastewater remediation.

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